FIRE CHARACTERIZATION OF AIRCRAFT GRADE POLYMERIC MATERIALS

Arthur F. Grand, Ph. D.
Omega Point Laboratories, Inc.
Elmendorf, TX USA

ABSTRACT

Conflicting results from different fire test methods on polymeric materials may be due to factors such as specimen orientation, specimen size, imposed heat flux, and method of calculating results. Observations of the burning characteristics of the specimens – including melting, char formation, and thermal stability – also are important to understanding the data from various fire test methods. Studies of aircraft-grade materials using the cone calorimeter will be used to illustrate the problems in characterizing the fire test performance of intumescent (char-forming) materials. Comparisons of results between the cone and OSU heat release rate calorimeters may be dependent on char formation and other burning characteristics of the specimens.

INTRODUCTION

The fire properties of materials or products inside commercial aircraft have a direct bearing on the potential spread of fire inside the cabin during a survivable post-crash fuel fire and, therefore, on the escape time for passengers of the plane. Unfortunately, the fire properties of materials and products are not inherent to those materials, but are dependent on the fire test methods used to develop the results. Thus, selection of test methods and interpretation of the results from such protocols must be performed diligently.

The Ohio State University (OSU) apparatus was selected by the FAA in 1984 for evaluation of the heat release rate (HRR) properties of interior wall and ceiling materials. The decision to use heat release rate to evaluate materials was a good one, and the OSU test method was selected for a variety of good reasons, including the following:

- the specimen is subjected to an external heat flux in the presence of an ignition source;
- screening is conducted on laboratory-size specimens; and
- the results of this test have relevance to full scale results.

Following development of the OSU apparatus, several other laboratory, intermediate and large scale tests test methods have been developed which are also based on evaluation of heat release rate. Since HRR is probably the most significant fire property that can be measured, it is important to understand the relationships among the various test methods and to learn how to interpret the results from these methods. In particular, it is still not clear whether or not one can obtain similar results from the same materials evaluated under different HRR test methods. As a consequence, the utility of the results from these methods for application to fire hazard analysis remains uncertain.

The results and data analysis presented herein are part of a Phase II SBIR study for the U. S. Department of Transportation. The title of the study is “Innovative Fire Retardant Polymeric Systems,” and methods of analyzing and interpreting HRR results have become a natural by-product of the program.

DESCRIPTION OF TEST METHODS

Most of the laboratory studies reported herein were conducted using the cone calorimeter (“cone”), as described in ASTM E1354 and ISO 5660. Other HRR test methods that will be discussed in this paper include the Ohio State University (“OSU”) and the intermediate scale calorimeter (“ICAL”). The most recent version of the OSU apparatus and test method are described in the FAA Aircraft Materials Fire Test Handbook; older versions are documented in FAR 25.853, and in ASTM E906 which is currently undergoing revision to be in agreement with the FAA method. The ICAL is described in ASTM E1623 (currently undergoing revision) and in ISO TR 14696. These three test methods were developed to obtain information on the ignitability and heat release rate characteristics of specimens under realistic fire test conditions. Each was designed to measure the HRR characteristics of samples of actual products, although they are limited to flat specimens. Each has advantages and disadvantages for various uses; however, only the OSU results are presently acceptable by the FAA for qualification of materials for the interiors of aircraft. The other two methods are used primarily for research and development studies (although they are and will be used for regulatory applications outside the FAA).

Some important characteristics of the three fire test methods, including certain advantages and disadvantages, are summarized below:

Cone – Specimens for the cone calorimeter are generally 100 mm (3.9 inches) square, although unusual size specimens can be tested. This small size is advantageous for product research and development investigations; however, the relatively large edge-to-surface-area ratio may be a factor for certain specimens where edge burning is a significant factor. While specimens can be exposed either vertically or horizontally in the cone calorimeter, nearly all tests are conducted with the specimen in a horizontal configuration. This avoids any artifacts due to melting or sagging of the specimen during the test, and permits adjustment of the distance between the surface of the specimen and the bottom of the radiant heater in order to avoid problems due to intumescent char formation. This is a distinct advantage for the cone calorimeter for such specimens, as will be discussed below. The ignition source in the cone is a spark igniter, which does not affect the heat flux at the surface of the specimen and is an improvement over the OSU ignition protocol. Perhaps the greatest advantage of the cone calorimeter over the other two test methods is the ability to operate at external heat fluxes up to 100 kW/m^2. A jet fuel fire can impose heat fluxes well in excess of the 35 kW/m^2 specified for the OSU test method and it is important to be able to study the impact of higher fluxes on test specimens.

OSU – This method is the only HRR method accepted by the FAA for certification of certain interior aircraft materials. The specimen must be 150 mm (6 inches) square and is supported vertically in a metal frame. It is exposed to an external radiant heat flux of 35 kW/m^2 (3.5 W/cm^2). While this is an adequately high heat flux for screening purposes, the apparatus is essentially limited to this heat exposure, which is a disadvantage for research and development studies. An ignition flame directed at the specimen generates a higher flux at the point of impingement, also a limitation in comparison to the other methods. Heat release rate is determined through thermal measurements, which is considered "older" technology compared to the method of oxygen consumption calorimetry employed by the cone and ICAL. Also, thermopile measurements account only for the convective heat released by the specimen, missing the radiant heat component of the total HRR, as pointed out by the developer of the apparatus (Smith, 1995).

ICAL – The main advantage of the ICAL method is in its specimen size. The 1 m (39 inch) square specimen is closer to the size of a real product than the specimens used for the cone or OSU, if that is an important consideration. However, the method is limited to vertical testing, which creates potential influences from the specimen melting, sagging or falling out of the frame. The ICAL is presently limited to a maximum heat flux of 50 kW/m^2, adequate for most studies but not as high as for the cone calorimeter.

MATERIALS

In this SBIR program, most of the studies were performed with Ultem® 9075, a modified polyetherimide (PEI), or on this PEI with various additives. The base material was selected from amongst several candidates partly because it was a thermoplastic compound (so we could melt-blend additives into it), and partly because we considered it to be a candidate for improved fire performance, even though it is an acceptable material under current FAA standards. Certain aspects of our work with this compound have been described by Weil et al. (1998), Grand and Bundick (1998) and Grand and Weil (1998). Some recent studies were conducted with Radel® R, a polyphenylsulfone (PPSU). Both of these materials are currently used in aircraft interiors. Additional thermoplastic and thermoset materials also used for aircraft applications are presently being tested as part of this program.

RESULTS AND DISCUSSION

We have evaluated a large number of additives to the base PEI compound at 50 kW/m^2 heat flux in the cone. Most of the additives did not demonstrate any significant improvement in the fire performance characteristics of the base resin. The data were evaluated mostly in terms of times to ignition and peak heat release rates. In retrospect, it seems that the intumescent (i.e., char forming) characteristics of the PEI compound may have overwhelmed any incremental effects of the additives. As a result, the repeatability of tests on the base compound was not as good as desired, thus affecting the comparisons between the base resin and any additive formulation. As will be illustrated below, the peak HRR values may not necessarily be the best indicators of fire performance.

As another consequence of experimental hindsight, later in the program we sought and found another specimen mounting technique for intumescent materials. In this technique (Gensous and Grayson, 1996), a 60 mm separation between the top of the specimen and the bottom of the cone heater, rather than the usual 25 mm, is recommended. The additional distance between the specimen and heater permits the char to grow without risk of touching the spark igniter or the heater. This recommendation is part of the current ISO 5660 cone calorimeter method, but is not mentioned in ASTM E1354, when dealing with intumescent materials. The cone heater must be recalibrated to the desired heat flux at the 60 mm distance.

The repeatability of results on intumescent specimens is not necessarily better at the 60 mm separation, compared to 25 mm (Grand and Weil, 1998). However, the technique is simpler and operator variability is reduced. (With the 25 mm separation, the heater/spark assembly has to be raised during a test with intumescent materials to prevent the char from touching either the sparker or heater).

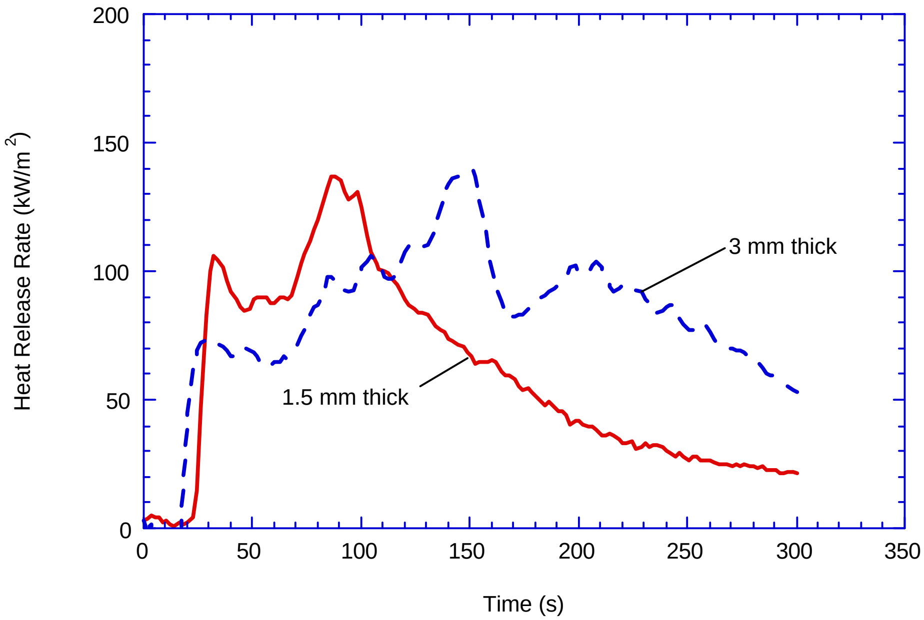
Recently, we obtained results on the PEI and PPSU blanks at different specimen thicknesses and different heat fluxes in order to compare the effects with ordinary, non-intumescing materials. Results for the PEI are shown in Table 1. We compared times to sustained ignition (t_{ig}), peak heat release rates (pk HRR), 60-second average heat release rates (avg HRR) and effective heats of combustion (Hc_{eff}) for the five minute test duration. Also, we considered the time to the peak HRR (t_{pkHRR}). The values shown are averages of two or more individual runs.

TABLE 1. SUMMARY OF RESULTS ON PEI COMPOUND AT DIFFERENT THICKNESSES AND HEAT FLUXES

Ht Flux, kW/m²	**Nom. Thick, mm**	**t_{ig}, s**	**Pk HRR, kW/m²**	**Time to Peak, s**	**Avg HRR, kW/m²**	**5-min. THR, kJ**	**Avg. Hc_{eff}, MJ/kg**
50	1.5	70	105.5	78	48.7	107	18.3
50	3	146	48.8	205	21.2	92	17.5
75	1.5	29	136.9	82	108.7	185	22.9
75	3	24	135.3	156	68.1	233	22.1

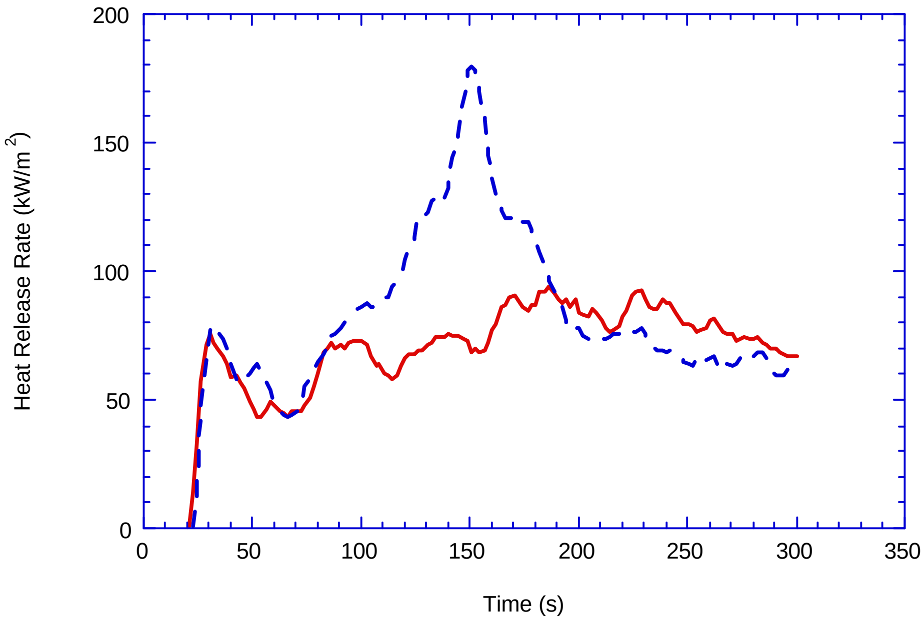
With the PEI blanks at 50 kW/m^2, increasing the thickness from a nominal 1.5 mm (1/16 in.) to approximately 3 mm (1/8 in.) produced longer t_{ig}, lower pk HRR (and lower avg HRR), but similar Hc_{eff} values. These effects are in the direction one would expect when testing thicker, non-intumescent specimens. On the other hand, at 75 kW/m^2, moving to the thicker specimen produced almost no change in t_{ig}, the same pk HRR values (but lower avg HRR), and similar Hc_{eff} (although the Hc_{eff} at 75 kW/m^2 was higher than that at 50 kW/m^2). For these specimens, the 60-s average HRR values may be a better comparative measure than the peak HRR. The "maximum" value is not necessarily the first peak because there are a series of peaks and valleys in the HRR plot. This can be seen from the comparative times to peak HRR in the table and from the appearance of representative HRR plots (e.g., in Figure 1).

FIGURE 1. HEAT RELEASE RATE–TIME PLOTS FOR PEI AT TWO DIFFERENT THICKNESSES AND 75 kW/m^2 HEAT FLUX



In this figure, the increase in thickness of the specimen can be seen to extend the time at which the peak HRR occurs. However, the presence of a maximum value so far into the experiment makes the reliability of that measurement questionable. In Figure 2, replicate experiments on PEI specimens demonstrate even more dramatically the HRR artifacts later in the run.

FIGURE 2. HEAT RELEASE RATE–TIME PLOTS FOR REPLICATE TESTS ON PEI AT 75 kW/m^2 HEAT FLUX



The "peak" HRR values shown in this figure are very different from one another, but the two runs had essentially identical 60 s average HRR. The extensive char from this material under 75 kW/m^2 heat flux, reached the bottom of the cone heater 60 mm from the original surface of the specimen. During this char formation, small flames popped out of the char like jets on the side of an active volcano. The HRR results are therefore somewhat erratic. Except for the earliest portion of the test run, flaming does not cover the surface of the specimen.

Given the extensive char formation, we decided it would be impractical, if not impossible, to run these materials in a vertical configuration in the cone calorimeter where the specimen-cone separation distance is fixed at 25 mm. The char formation must be considerably lower in the OSU apparatus under the usual 35 kW/m^2 heat flux conditions of that test, compared to the higher fluxes used in the cone; otherwise, the char would interfere with the action of the igniter in the OSU test.

Earlier this year, comparisons of OSU and cone results were presented for PEI and other materials (Grand and Bundick, 1998). Even using cone data at 50 kW/m^2, comparisons to the OSU were not particularly good for these materials, most of which were resistant to sustained burning even at 50 kW/m^2 heat flux. Actually, the peak HRR values for PEI specimens in the cone at 50 kW/m^2 and the OSU at 35 kW/m^2 were comparable, but the appearance of the HRR-time curves – and therefore the patterns of burning – were very different. OSU-cone comparisons for a series of non-intumescent, fire-resistant wire compositions (Schwartz et al., 1998) produced a consistent pattern in peak HRR values between the cone (at 50 kW/m^2) and the OSU (at 35 kW/m^2). It is the opinion of this author that materials with even burning characteristics, whether or not they burn readily under the OSU test conditions, have the potential to generate a correlation with cone calorimeter test results conducted at a higher flux. However, for intumescent materials, and possibly for others that do not burn regularly, a correlation probably does not exist. Furthermore, as Smith (1995) pointed out, specimens generating large quantities of smoke probably do not produce correct HRR results in the OSU apparatus; therefore, the likelihood of developing correlations with cone calorimeter results for such specimens is reduced further.

A discussion of why 50 kW/m^2, or higher, for the cone is required to compare to 35 kW/m^2 in the OSU is appropriate. Most likely, the impinging pilot burner in the OSU creates a higher, localized heat flux near the bottom of the specimen. For any specimen that might burn under these OSU test conditions, the HRR will be characteristic of the higher heat flux around the igniter. Also, the cone heater is regulated so that it reduces its radiant heat output as the specimen burns. In this way, the actual heat flux to the surface of the cone specimen is constant (otherwise, the surface flux is the sum of the initial flux and that created by the flaming specimen). There is no such system in the OSU. Therefore, any burning by a specimen at the site of the igniter will affect the remainder of the specimen in proportion to its heat output.

A summary of recent tests on PPSU specimens in the cone calorimeter is presented in Table 2.

TABLE 2. SUMMARY OF RESULTS ON PPSU SPECIMENS AT DIFFERENT THICKNESSES AND HEAT FLUXES

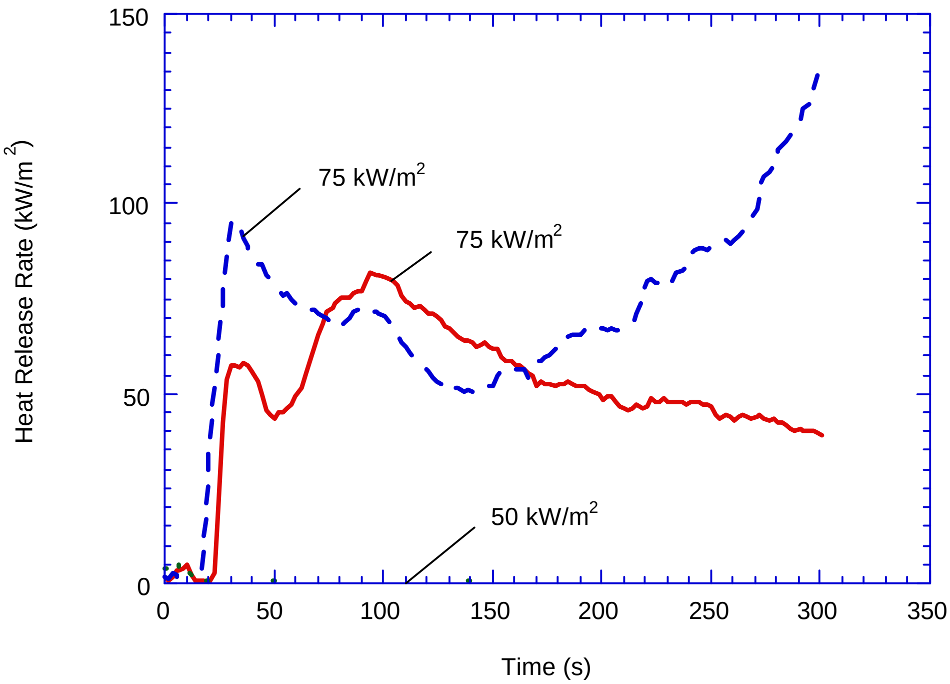
I. D.	**Nom. Thick, mm**	**Ht Flux, kW/m^2**	**t_{ig}, s**	**Pk HRR, kW/m^2**	**Time, s**	**Avg HRR, kW/m^2**	**THR, kJ**	**Avg. Hc_{eff}, MJ/kg**
PPSU-1	2	50	>168*	9.6	84	3.6	3	n.a.
PPSU-1	2	75	26	89.0	64	66.9	178	19.1
PPSU-2	1.5	75	34	112.9	36	86.8	197	19.7
PPSU-2	2	75	39	125.4	43	90.6	213	20.4
PPSU-2	3	75	44	128.8	45	57.4	106	18.3

*Shown is the average of the specimens that ignited, several did not ignite.

The PPSU specimens did not burn easily or regularly under the conditions of 50 kW/m^2 heat flux in the cone. When ignition did occur, it was late in the run and the peak HRR values were generally 10 kW/m^2 or less (for these specimens, OSU peak values were also less than 10 kW/m^2). At 75 kW/m^2, more repeatable ignition and burning occurred. Similar to the PEI, these specimens developed extensive chars. Also, there were small changes in the measured parameters as functions of thickness. The 60 s average HRR values changed little from 1.5 to 2 mm, with a more noticeable change evident at the 3 mm thickness.

Plots of three test runs on PPSU, one at 50 kW/m^2 and two at 75 kW/m^2, are presented in Figure 3. With no ignition at the lower flux, essentially zero heat release was detected. This produced a marked contrast to the results at the higher flux. At 75 kW/m^2, the two curves shown have rather different appearance. This is an effect of the irregular burning characteristics. In one case, the maximum HRR value is the first "peak" in the curve, while in the second case the maximum is the second "peak." Curiously, the maximum HRR values are not too different from one another (96 and 82 kW/m^2); while the 60 second average HRR numbers are farther apart (76 and 58 kW/m^2, respectively). It remains to be determined whether the 60 s average values might be more reliable than the peak values, as it seemed for the PEI specimens.

FIGURE 3. HEAT RELEASE RATE–TIME PLOTS FOR TESTS ON PPSU AT 50 AND 75 kW/m^2 HEAT FLUX



Our intent, as part of this study, was to perform testing using the intermediate scale heat release calorimeter, the ICAL. Given the lack of a promising correlation between the OSU and the cone for selected specimens (Grand and Bundick, 1998), it seems that another test method should be included in the correlation matrix. We wondered whether this device could be the missing link in HR correlations to larger scale, or whether it would simply create another set of questions.

The tests conducted to date using the ICAL, on a thermoplastic sheet material on an aluminum substrate, were not conclusive. Under both the cone calorimeter and OSU at 35 kW/m^2, the specimen ignited and burned, producing a peak HRR of approximately 60 kW/m^2 in both the cone and the OSU. In the ICAL at 50 kW/m^2, one specimen did not ignite, while another ignited and produced a peak HRR of a similar order of magnitude as the cone and OSU experiments. There was no melting or other unusual effects that would compromise the data from this setup.

CONCLUSIONS AND RECOMMENDATIONS

One of the objectives of this overall program is to characterize materials in such a way that the results of the fire performance test protocols can be used in an evaluation of the potential fire hazard of the materials. Right now, the difference between HRR values for any two materials is only a number. It has not been quantified in terms of an increase or decrease in the potential threat to the occupants of an aircraft.

Heat release rate evaluations on aircraft-grade materials should be incorporated into an estimation of fire hazard. Only then will we be able to quantify the impact of material flammability performance on human escape time in a survivable post-crash fuel fire. The HRR information should be obtained by any means available, including bench scale (cone and OSU), intermediate scale (ICAL) and room scale evaluations of wall lining materials.

For intumescing materials, additional consideration must be given to the methodology and specimen exposures, compared to materials with ordinary flammability characteristics (even materials that do not burn readily). While it is not a straight-forward task to evaluate HRR properties of intumescing specimens in the cone calorimeter, this method at least offers the opportunity to change the test conditions to accommodate the unique properties of such materials. The OSU heat release rate apparatus, while adequate for screening many materials, probably is not suitable for meaningful testing of intumescent products.

It has been suggested that a lower heat release rate and higher heat flux exposure should be the goal for aircraft grade interior materials. Specifically, a target of zero heat release rate at 50 kW/m^2 heat flux, compared to the present 65 kW/m^2 peak HRR at 35 kW/m^2 heat flux, has been recommended. In view of the fact that 50 kW/m^2 in the cone is required to compare to 35 kW/m^2 in the OSU, we should be concerned about whether a heat flux of 50 kW/m^2 in the cone is adequate. In addition, fire hazard modeling should be implemented to support a lower HRR goal.

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